

X-Ray Characterization of 3-Layer Assembly of 4 nm FePt Nanoparticles

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Scientists from IBM's Thomas J. Watson Research Center in Yorktown Heights, New York and Almaden Research Center in San Jose, California have developed a chemical reduction process for making monodisperse 4 nm FePt nanoparticles. Further, they used polymer-mediated self-assembly approach to assemble the 4 nm FePt nanoparticles on various substrates. By using X-ray diffraction (XRD) in grazing incidence geometry at beamline X20C, the scientists have determined the structure of the assembly and the particles, and studied the aggregation behavior of the particles in an assembly of 3 nanoparticle layers under thermal annealing conditions.



The authors (from left, top): Thomas Thomson, Simone Anders, John E.E. Baglin, Bruce D. Terris, and Mike F. Toney, (bottom) Shouheng Sun, C.B. Murray, and Hendrik F. Hamann

Hard magnetic FePt nanoparticles have attracted great interest because of their potential applications in ultrahigh density magnetic recording, highly sensitive magnetic sensors, and advanced nanocomposite permanent magnets. Recent advances in magnetic recording technology have indicated that if self-assembled in a tightly packed, exchange-decoupled array with controlled magnetic easy axis direction, these FePt nanoparticles would be a candidate for future ultrahigh density data storage media with potentially one bit per particle.

We report a simple chemical process for synthesizing FePt nanoparticles by the reduction of FeCl_2 and $\text{Pt}(\text{acac})_2$. The particle growth is self-limited and 4 nm FePt nanoparticles are readily separated. The initial molar ratio of the metal precursors is carried over to the final product, and the FePt composition is easily tuned. We further demonstrated that alternate adsorption of polyethylenimine (PEI) and FePt nanoparticles on a hydroxyl (HO)-terminated surface via surface ligand exchange led to 4 nm FePt nanoparticle assemblies with controlled thickness. **Figure 1A** illustrates the general assembly approach.

We characterized the layered structure using X-ray reflectivity measurements using $\text{CuK}\alpha_1$ radiation from an 18 kW X-ray generator. X-ray reflectivity measures the electron density of the nanoparticle assembly, which can be converted into mass density. The result of this analysis for a 3-layer assembly is shown in **Figure 1B**, which plots mass density as a function of position from the silicon substrate surface. The three layers are readily evident as regions of larger mass density than the PEI between the nanoparticle layers. The spacing between both the first-second and second-third layers is 6.5 nm.

We studied the structure of the FePt nanoparticles in a thin assembly using X-ray diffraction (XRD) measurements performed in grazing incidence geometry at beamline X20C. As shown in **Figure 2**, the XRD of the as-synthesized FePt particles reveals a typical chemically disordered fcc structure, in which Fe and Pt atoms randomly occupy the fcc lattice sites. Thermal annealing induces the Fe and Pt atoms to rearrange into the chemically ordered face-centered tetragonal structure,

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which can be viewed as a natural superlattice of alternating Fe and Pt atomic planes along the (001) direction (**Figure 2**). From the line width of the (111) peak of the fct ordered FePt, we have also estimated the (111) coherence length that is related to the particle size. The average particle size increases with annealing temperature and duration. The particle size estimated from XRD line width for 3 layer assemblies (**Figure 2**) is 5 nm for the assemblies annealed at 580°C for 30 minutes, but rises to 17 nm for those annealed at 800°C for 5 minutes. The work demonstrates that synchrotron radiation is a powerful characterization tool for thin (~ 10 nm) FePt nanoparticle assemblies.

Additional Publication:

S. Anders, M.F. Toney, T. Thomson, R.F. Farrow, J.-U. Thiele, B.D. Terris, S. Sun, and C.B. Murray, "X-ray Absorption and Diffraction Studies of Thin Polymer/FePt Nanoparticle Assemblies," *J. Appl. Phys.*, 93, 6299 (2003).

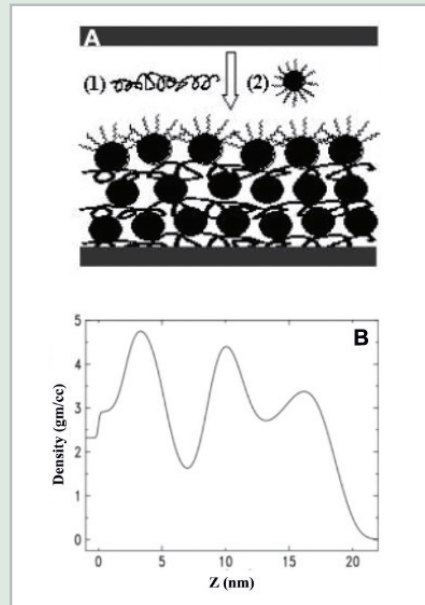


Figure 1. (A) Schematic illustration of polymer-mediated self-assembly of FePt nanoparticles by alternately adsorbing a layer of polymer (PEI) and a layer of nanoparticles on a solid surface; and (B) Mass density of a 3-layer FePt assembly, deduced from the X-ray reflectivity measurement, as a function of position from the silicon substrate (z). The silicon surface is arbitrarily defined as $z = 0$.

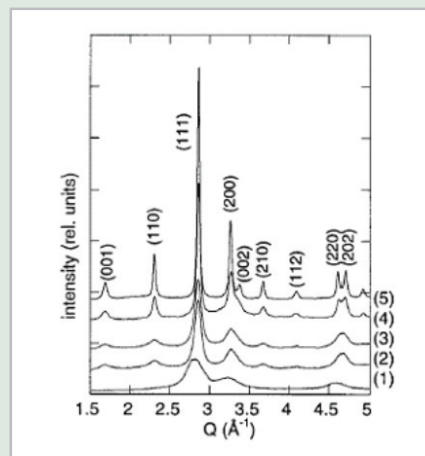


Figure 2. In-plane X-ray diffraction of 3-layer $\text{Fe}_{58}\text{Pt}_{42}$ assemblies as function of annealing: (a) as deposited, (b) 580°C for 30 min, (c) 650°C for 5 min, (d) 700°C for 5 min, (e) 800°C for 5 min. The ordinate is the scattering vector Q , which is the difference between incident and diffracted X-rays. It has magnitude $Q = (4\pi/\lambda) \sin \theta$, where λ is the X-ray wavelength (about 0.12 nm here) and θ is half the scattering angle. The index of diffraction peaks is marked.